High-Temperature Delamination Mechanisms of Thermal Barrier Coatings: In-situ Digital Image Correlation and Finite Element Analyses

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Keywords: thermal barrier coating; thermal damage mechanics; digital image correlation;
finite element analysis

Abstract

Through *in-situ* high-temperature digital image correlation (DIC) and finite element simulation, we unveil new, critical delamination and fracture mechanisms of dense vertically cracked thermal barrier coatings (DVC TBCs). Full-field DIC strain maps of cross-sectioned TBCs during thermal cycling up to 1200°C reveal the coupled formation mechanisms of vertical and horizontal cracks, which showed that horizontal cracks were formed by vertical crack deflection/branching. Horizontal crack growth was characterized by crack bridging, crack shielding, and crack deflection. Internal defects and weak interfaces such as pre-existing voids and inter-splat boundaries promoted crack formation. Finite-element simulations revealed that the interaction between the vertical and horizontal cracks produced favorable strain energy release rates (ERR) for horizontal crack initiation and propagation on the interior of the coating compared to along the bond coat interface, which contrasts with conventional delamination

mechanisms. These results indicate that high-temperature DIC is a powerful tool for *in-situ* measurement of microstructure-processing-deformation-failure relationships that are otherwise unobtainable.

1. Introduction

Thermal Barrier Coatings (TBCs) are commonly used to increase the durability and operating temperatures of gas turbines, with the goal of improving engine efficiency and reducing operating cost [1,2]. Typically, these coatings are multilayered, such as the commonly used Yttria-stabilized Zirconia (YSZ) top coat (TC) / NiCoCrAlY intermetallic bond coat (BC) / superalloy substrate TBC system. Each of the constituent layers has different thermal and mechanical properties, resulting in a complex strain/stress state during thermal cycling. Moreover, the coating interfaces are generally rough, and the coatings themselves contain defects and are highly porous [3]. This nonlinearity makes robust numerical simulation difficult and fully-detailed analytical solutions all but impossible. To date, there has been no *in-situ* study to quantitatively relate crack initiation or propagation with respect to the microstructure, which is essential to validating the boundary conditions and results of finite-element (FE) TBC failure simulations. Recent synchrotron X-ray approaches have only partially solved this problem; they simply capture lattice strains without regard to microstructure using high-temperature synchrotron X-ray Diffraction (XRD) [4,5] or alternately capture deformation as a function of microstructure with room-temperature *ex-situ* synchrotron X-ray computed tomography (XCT) [6]. These measurements reveal a nonlinear stress pattern through the coating thickness, but it is not clear how strain relates to microstructure *during thermal cycling*.

The failure mechanisms of TBC systems have been widely studied with both FE and post-failure microscopy techniques. Remarkable scientific progress has been achieved by these

methods with regards to interfacial delamination of TBCs, which generally occurs between the coating and bond coat or coating and substrate [7–10]. However, these models and experiments assume a *homogeneous ceramic top coat without surface cracks*. It has recently been established through FE simulations that small vertical or horizontal cracks near the TC / BC layer interface can reduce tensile stresses in the thermally grown oxide (TGO) layer and inhibit delamination [11]. Periodically spaced surface cracks, which penetrate the top coat from the free surface to the TGO interface, increase coating compliance, and enhance interfacial durability [12,13]; indeed, this is the motivating factor for so-called “Dense Vertically Cracked” (DVC) TBCs. It can be expected that surface cracks may significantly alter the failure mechanisms of TBCs in ways that have not been studied.

Second, the role of microstructural variation on TBC failure is not well understood. Coatings deposited by air plasma spray (APS) contain a microstructure with many pores, cracks, and inter-splat voids [3], which are potential failure initiation sites. Some of these features, in particular the inter-splat voids, are oriented parallel to the TC/BC interface, and large defects may redistribute stress away from the TGO and inhibit delamination near the TC/BC interface [11]. This may contribute to an alternative delamination mechanism.

In the current work, a DVC TBC system is shown to exhibit vertical cracks from the TC surface to the TGO interface, and horizontal cracks that branch off of the vertical crack (Fig. 1). The two types of cracks are shown to interact and prevent delamination along the TC / BC interface. Instead, the horizontal crack is shown to grow with repeated thermal cycling at temperatures up to 1200°C and eventually lead to coating failure. Through this comprehensive study, we establish a new TBC failure mechanism of delamination in the middle of the TC thickness. First, polished cross-sections of as-deposited TBC samples were thermally cycled

from 25°C to a maximum temperature between 1000°C and 1200°C under an optical microscope and analyzed with high-temperature digital image correlation (DIC) techniques [14]. Full-field thermomechanical deformation results are then related to (a) crack growth measurements and (b) salient microstructural features. Experimental results are reported in Section 3. Second, a high-throughput FE-based fracture analysis scheme elucidates the cracking mechanisms observed in the DIC experiments. This approach incorporates the effects of TC / BC interfacial roughness, uses experimentally measured high-temperature mechanical properties and systematically varies the horizontal crack position and crack length to understand the crack driving forces. This modeling approach is somewhat similar to recent work by Richards [15], with the important distinction of incorporating experimentally measured interface geometries and nonlinear material models, which are shown to strongly influence the failure behavior of TBCs [8,16,17]. Fracture analysis results are reported in Section 4.

2. *Experimental Methods*

Thermal cycling experiments were performed on TBC samples with representative coating materials and thicknesses (Table 1). The coating system consisted of an 8%_{wt} YSZ (8-YSZ) top coat, a NiCoCrAlY bond coat and a Haynes HR-224 substrate. The as-deposited coatings were sectioned into ~2 mm x 10 mm sections and then polished with standard metallographic techniques. During thermal cycling, a thin TGO layer grew on the BC layer.

2.1. *Thermal Cycling Experimental Methodology*

To study the crack initiation and propagation mechanisms, polished TBC cross-sections were thermally cycled in an optical microscope heating stage (Linkham Scientific, TS1500) and simultaneously imaged at 20x magnification. Five samples were heated (30°C / minute) to a maximum temperature of 1000, 1050, 1100, 1150, and 1200°C, respectively, held at this

temperature for 60 minutes, and then cooled to a minimum temperature of 100°C (also at 30°C / minute); this heating cycle was repeated for a total of 4 cycles per sample. An additional sample was thermally cycled to a maximum temperature of 1200°C for 80 cycles to study crack propagation and failure.

Digital images (5 Megapixel, PointGrey Research) of each sample were acquired before cycling, as well as at 100°C, 600°C, and at the maximum temperature during both the heating and cooling portions of the loading cycle. Ultraviolet lighting and narrow UV bandpass filters (450±20 nm wavelength) enabled high-contrast imaging of the TBC sample elevated temperatures, which was necessary for successful image correlation [14]. After using commercial focus-stacking software to create a fully-focused image, the stitched images were correlated with commercial DIC software (Vic-2D, Correlated Solutions) to extract full-field local strain measurements throughout the TC cross-section. The TC's intrinsic porosity provided a suitable high-contrast surface pattern for accurate correlation [18]. The area of interest was carefully defined to exclude the TGO and BC layers, which would otherwise introduce bias to the strain measurements. The resulting DIC dataset was used to identify cracking and other failure events, as the crack opening displacements produced large pseudo-strains. To isolate mechanical deformation and crack-opening strains, thermal expansion relative to the reference state was subtracted from the measured DIC strain fields using literature thermal expansion coefficients ($12.0 \mu\epsilon \text{ } ^\circ\text{C}^{-1}$ for TC) [19,20]. Finally, it is emphasized that DIC's subpixel resolution enables highly-sensitive detection of deformation and cracking, even if microscopic crack opening is not visible in the image.

DIC error associated with thermal convective currents and focus stacking was evaluated by correlating several images acquired at 1200°C. As a measure of error, the standard deviation

of the computed artificial strains was found to be $\Delta\epsilon_{xx} = .021\%$ and $\Delta\epsilon_{yy} = .041\%$. Error in ϵ_{yy} was random, while error in ϵ_{xx} show undulatory behavior with the largest error associated with image stitching boundaries; this error was attributed to overlapping regions with poor focus [21]. In all cases, this error was found to be an order of magnitude below crack-opening pseudo-strains and did not have a significant effect on strain calculations.

2.2. *High-temperature Mechanical Properties through Nanoindentation*

To assist with FE analysis, the high-temperature mechanical properties of the coating system were obtained by nanoindentation. The elastic modulus from 25-1000°C was obtained from indentation loading/unloading curves using the Oliver-Pharr method [22]. Indentations were performed on a nanoindenter (MicroMaterials Vantage) and a tribometer (Bruker Nano UMT 3) using a cubic boron nitride (cBN) Berkovich indenter and a sapphire Vickers indenter respectively. Individual grains in the TC and BC were targeted for nanoindentation with loads of 50 and 100 mN, which was found to reduce measurement variation. Calculations assumed $\nu_{cBN} = 0.12$, $E_{cBN} = 800 \text{ GPa}$, $\nu_{sapphire} = 0.24$, $E_{sapphire} = 375 \text{ GPa}$ at room temperature [23] and indenter mechanical properties were corrected for elevated temperature dependence to conform with reported temperature dependence in literature [24,25]. To compute TC fracture toughness, an array of 15 indentations at a peak indentation load of 500mN was performed at 25°C on both untreated and 1200°C-treated (40 cycles) specimens. SEM measurement of the resulting radial cracks was used to compute fracture toughness (K_{IC}) using the relationship,

$$K_{IC} = B \sqrt{\frac{E_Y}{H}} \frac{F}{c^{3/2}} \quad (1)$$

where B is an indenter geometry constant, E_Y is the Young's Modulus, H is the hardness, F is the applied load, and c is the crack length [26,27].

Indentation results are briefly summarized in Fig. 2, which maps E_Y as a function of

temperature and position in each TBC material. The stiffness of each layer decayed linearly up to 1000°C (Fig. 2a); for example, the modulus of the TC was 58.6 GPa at 25°C and 41.9 GPa at 1000°C, which agreed closely with literature values [28]. The elastic moduli obtained by nanoindentation varied significantly from layer to layer (Fig. 2b), resulting in severe gradients at layer interfaces. The material adjacent to the TGO/BC and BC/Substrate interfaces exhibited low stiffness for unexplored reasons, possibly due to compositional gradients or interfacial defects. Moderate modulus variation was also observed within individual layers due to stochastic microstructural effects (voids, cracks and other defects) [29,30]. Elastic moduli for each layer were linearly extrapolated to 1200°C to provide mechanical properties for FE simulations. The average calculated TC fracture toughness of the untreated TBC was $K_{IC} = 1.77 \text{ MPa m}^{1/2}$ while the average toughness of the 1200°C-treated TBC specimen was $K_{IC} = 1.46 \text{ MPa m}^{1/2}$; converted to ERR, the two fracture toughnesses were $G_{IC} = 53.5$ and 36.4 J m^{-2} , respectively. These values were on the lower end of ranges presented in other literature [31–33]. The 17.4% decrease in K_{IC} after thermal cycling suggested that continued exposure to ultra-high temperatures and material oxidation significantly reduces the fracture resistance and facilitates crack propagation. We emphasize that the indentations targeted dense material in the TC, meaning that these experiments provided upper bounds for G_C and that crack growth along weak interfaces could be substantially easier.

2.3. FE Model Development

Several FE simulations were performed to understand the fracture mechanisms of the TBC sample. Separate models with flat and experimentally-measured interface geometries were developed, with the coating thickness and interface geometry (Fig. 3) established from optical micrographs and X-ray computed tomography (Xradia microXCT 200). The rough interface was

irregular with roughness $R_a = 16.2 \text{ }\mu\text{m}$, a maximum peak-to-valley distance of $69.7 \text{ }\mu\text{m}$, and a characteristic wavelength of $\approx 200 \text{ }\mu\text{m}$. The interfaces between coating layers were modeled with shared nodes, since interfacial delamination was not of interest to these simulations.

The central objective of the simulations was to characterize the initiation and propagation of horizontal cracks from the vertical crack. To achieve this, a vertical crack was positioned at an experimentally accurate location near a “valley” in the TC/TGO interface, and a horizontal crack of length L branched off of the vertical crack at a depth d from the top surface. Both d and L were manipulated over the range $d \in (25, \dots, 230 \text{ }\mu\text{m})$ and $L \in (10, \dots, 250 \text{ }\mu\text{m})$ to fully map the crack growth characteristics as a function of microstructure. Material models were developed for each of the coating layers with both experimental (Section 2.2) and literature values for material properties, as summarized in Table 2. The bond coat material used a linear elastic / perfectly plastic material model based on prior literature values [34]. The nominal element size away from the crack tip was $3.75 \text{ }\mu\text{m}$, and the mesh was refined to a size of $0.08 \text{ }\mu\text{m}$ near the crack tip (Fig. 3b). Each model consisted of approximately 13500 nodes and 13250 elements, of which 1700 nodes and 1700 elements were located in the refined crack tip mesh. Mesh generation was automated with a Python script, facilitating high-throughput fracture analysis of the TBC failure mechanism.

The FE models were subjected to ramped temperature profile between 25°C and 1200°C with a reference temperature of 25°C , as well as from 1200°C to 25°C with a reference temperature of 1200°C . These two conditions approximated the sample’s first annealing after coating deposition in which the sample is approximated as initially free of residual stress and cool down after stress relaxation at maximum operating temperature. Since the material models were independent of time, these simulations neglected creep and stress relaxation effects; the

temperature profiles should be interpreted to have rapid heating and cooling rates to prevent viscoplastic effects. The crack driving force in each model was evaluated with the virtual crack closure technique, which enabled the computation of energy release rate at the crack tip [35]. Energy release rate was computed at every 25°C temperature increment in the loading profile, providing a deep understanding of the crack growth mechanism during thermal cycling.

3. *Thermal Cycling Results*

3.1. *Crack Initiation*

Temperature-corrected DIC strain maps acquired during the initial heating of the TBC samples revealed a heterogeneous strain distribution, indicative of localized cracking and microstructure-dependent deformation. Maps of ϵ_{xx} and ϵ_{yy} for the 1100°C sample are presented in Figs. 4 and 5 respectively, which are representative of strain fields for all 5 samples. The ϵ_{xx} strain field was periodic with an apparent wavelength of $\sim 200\ \mu\text{m}$, and the strain peaks spanned most of the TC thickness. Inspection of the underlying micrograph revealed the presence of DVC-type vertical cracks at each local maximum, implying that these strain fields were caused by crack opening pseudo-strains; growth of these cracks was driven by bulk tensile stresses in the TC when heating from a stress-free state (Fig. 4c) [18]. At 600°C, the magnitude of each local maximum was $\epsilon_{xx} \approx .025$; at 1100°C, the pseudostrain at the primary crack increased to $\epsilon_{xx} = .05$, while the magnitude of strain at the minor crack remained constant. Therefore, the growth of the primary vertical crack relieved the stress on adjacent vertical cracks and prevented further crack opening.

During the initial heating, horizontal cracks formed off of the vertical crack (Fig. 5), as marked by a pattern of ϵ_{yy} pseudostrains. Their initiation appeared as regions of elevated ϵ_{yy}

strain below 600°C, and crack opening displacement (COD) grew upon heating at 1100°C (Figs. 5b-c). This indicated crack formation at lower temperature and continued growth at elevated temperatures. High-magnification SEM analysis (Fig. 5d) confirmed the presence of lateral cracks and also showed that lateral cracks initiated at corners in the vertical crack, revealing that their formation was closely coupled with vertical crack deflection; these mechanisms are analyzed in more detail in Section 3.3. The initiation of cracks at lower temperatures was unexpected, given that thermomechanical stresses due to mismatched thermal expansion were low for small ΔT . This early crack initiation highlighted the importance of residual stress, pre-existing flaws and the elevated stresses around the crack tip during vertical crack formation in initiating the horizontal cracks.

3.2. *Crack Propagation*

While the samples were assumed to be stress-free before thermal cycling (reference temperature of 25°C), stress relaxation occurred at elevated temperatures to modify the reference temperature for residual stress models based on mismatched thermal expansion. This transition changed the thermomechanical stress field which drives crack growth and marked a change between “crack initiation” and “crack propagation” phases. This creep-driven transition was studied by tracking the median DIC strain (which is insensitive to crack opening) during 4 thermal cycles at temperatures between 1000°C and 1200°C, with results summarized in Fig. 6. Using the image acquired immediately upon reaching the maximum cycling temperature as the reference image, these plots indicated that a steady-state stress distribution in each sample was not achieved until after the fourth cycle (Fig. 6). Evidence of a strain-ratcheting effect was present in all five samples, with median ϵ_{xx} decreasing linearly to values between $\epsilon_{xx} = -.015$ and $-.025$ after four cycles, but thereafter achieved a steady-state strain distribution (Fig. 6b).

The median ε_{xx} in the 1200°C sample quickly reached a steady-state values of $\varepsilon_{xx} = -.02$ after the second cycle, while the median strain in the remaining samples continued to decrease for 4 cycles. Minimal deformation occurred during the first hour-long exposure at the maximum temperature (*i.e.*, $t = 1 \text{ hour}$) in Fig. 6a, indicating most strain accumulation occurred during the cooling portion of each cycle. While the median strain was larger for the 1100, 1150 and 1200°C samples compared to the 1000 and 1050°C samples, indicating that the ratcheting effect generally scaled with temperature, variation in sample roughness prevented robust conclusions about this trend. Indeed, the median strain of the 80-cycle 1200°C sample achieved a peak median $\varepsilon_{xx} = -.032$ after two hours but returned to an equilibrium strain of $\varepsilon_{xx} = -.02$; the difference in behavior compared to the other samples was attributed to higher interface roughness (compare Fig. 5 vs. Fig. 7).

Temperature-corrected ε_{yy} maps are presented in Fig. 7 for the 80 cycle test, which reveal the basic crack propagation mechanisms associated with DVC TBCs. Prior to 10 cycles (Fig. 7a-b), the ε_{yy} pseudostrains associated with crack propagation were concentrated about 50 μm below the surface (crack marked by * in Fig. 7b). In subsequent cycling, another region of crack growth (marked by † in Fig. 7d) about 50 μm above the TGO interface developed significant COD and became the most prominent region of crack growth by the 20th cycle (Fig. 7c-d). This location continued to experience the most growth through 80 cycles. These results suggested that the near-surface crack initially possessed critical ERR for crack growth, but both the natural stress field for long near-surface cracks as well as crack shielding effects from the near-interface cracks reduced ERR to subcritical values. The mechanism of this transition from one prominent crack formation to a second is analyzed further with FE simulations (Section 4.2). In both positions, the cracks angled gently toward the BC interface at an angle of $\sim 15^\circ$, suggesting that growth

toward the interface resulted in most favorable ERR for long cracks. Close inspection of the crack path (Fig. 7e) revealed that crack growth occurred through preferential linkage of pores and voids along this critical angle. Growth did not occur in other directions, even along paths with seemingly favorable microstructures for fracture, such as the chain of large pores along the top of Fig. 7e. The highlighted path exhibited widespread bridging, indicating that the complex APS microstructure possessed irregular fracture toughness and that voids adjacent to the crack path altered crack-tip stresses. Depending on these local stress states, the crack may propagate around or through the defect.

3.3. *Cracking Mechanisms*

SEM imaging of the TBC samples after 4 thermal cycles (Fig. 8) was used to analyze the crack initiation and propagation mechanisms. Regardless of cycling temperature, all lateral cracks branched off of vertical cracks (marked by red arrows in Fig. 8a-g) and appeared to originate at deflections in the vertical crack path, confirming that the initiation of vertical and horizontal cracks was strongly coupled. The location of horizontal cracks suggested that as vertical cracks were arrested at microstructural barriers, lateral microcracks branched off of the obstacle and formed lateral cracks; this is depicted schematically in Fig. 8h. The insensitivity of crack formation mechanism to temperature indicated that the lateral cracks formed at temperatures below the smallest cycling temperature ($<1000^{\circ}\text{C}$), which was consistent with the formation of vertical cracks and DIC results.

The longest horizontal cracks and most sample damage occurred in the 1000°C sample compared to samples cycled at higher temperatures. After 4 cycles, the 1000°C specimen exhibited surface and near surface damage (Fig. 8a) that was not observed in specimens cycled to higher temperatures. In conjunction with median-strain results presented in Fig. 6, this damage

pattern suggested that stress relaxation occurred slowly at 1000°C compared to higher temperatures, and that rapid stress relaxation inhibited the critical growth of lateral cracks. The severity of damage in the 1000°C sample during the early stages of thermal cycling indicated that per-cycle damage in the “crack initiation” regime was more severe than in “crack propagation”.

Finally, the SEM images revealed specific mechanism of crack propagation, showing that crack growth was highly irregular and regulated by microstructure. These images provided several examples of bridging, shielding, branching and deflection of cracks, which suggested that microstructural features both arrested primary crack growth and promoted secondary cracking adjacent or ahead of the crack tip at weak interfaces. These cracking mechanisms were essential to the formation a network of cracks, as the arrest of a particular lateral crack enabled the growth of adjacent cracks. At the same time, the interrupted cracking pattern also meant that the TC material retained strength and toughness; however, the increased coating compliance could substantially alter interfacial stresses and vulnerability to other damage mechanisms.

4. *Finite Element Analysis*

4.1. *Crack Initiation: Role of Interface Geometry*

Results of simulations to probe the crack driving force during the sample’s first heating are summarized in Fig. 9. Simulation results are presented for both experimental and flat interface geometries, which emphasize that ERR was a nonlinear function of crack position and temperature. At moderate temperatures below 850°C, cracks positioned near the TC / BC interface ($d > 170 \mu m$) as well as near the TC surface ($d < 100 \mu m$) experienced an elevated ERR compared to cracks at intermediate depths, and ERR increased monotonically with temperature. At temperatures above 900°C, the ERR became insensitive to increasing

temperature; this transition was attributed to the reduced yield strength of the BC layer, suggesting that plastic flow strongly limited the crack driving force at elevated temperatures.

Second, TC / BC interface roughness strongly influenced ERR and created very different crack growth behaviors near the interface. This was evident both quantitatively in the maximum calculated ERR as well as qualitatively in the ERR contour shape as a function of temperature and crack position. With the experimental geometry, the maximum ERR occurred at 1200°C at a position 50 μm above the TC / BC interface with $G_{max} = 43.0 \text{ J/m}^2$. With the flat geometry simulation, the maximum energy release rate occurred at 825°C on the TC / BC interface with $G_{max} = 54.5 \text{ J/m}^2$. Thus, it could be expected that the interface geometry governs the maximum energy release rate as well as the location and temperature at which it occurs. Compared to the experimental (rough) interface geometry, the flat interface at moderate temperatures (T between 500 and 900°C) increased the amount of material with enhanced ERR in interior of the TC, and reduced the amount of material with elevated ERR near the interface. In addition, above 900°C and with the flat interface geometry, the ERR was nearly uniformly distributed for all crack positions: crack growth on the interior of the TC therefore became competitive with crack growth along the TC / BC interface.

With respect to the experimentally-observed crack growth mechanisms, the flat interface geometry facilitated the initiation of cracks on the interior of the top coat layer. Since the distribution of large flaws was statistical, and ERR was shown to increase with crack length, the larger interior region of material with elevated ERR increased the likelihood that a critical ERR was achieved. Although the ERRs for $L = 10 \text{ }\mu\text{m}$ were below G_C , equivalent analyses with $L = 20 \text{ }\mu\text{m}$ showed larger ERR at all temperatures in the sample interior. SEM analysis of unheated TBC samples revealed crack-like flaws longer than 50 μm , which could increase ERR

in the sample interior to levels approaching G_C .

4.2. Crack Propagation

As the majority of experimental crack propagation occurred after annealing at high temperatures, the crack driving force was also simulated during cool down from a hot reference temperature of 1200°C. In Fig. 10, ERR is presented at $T = 25^\circ\text{C}$ as a function of crack position d and crack length L . Results are shown only for the flat interface, as it was impossible to simulate a purely horizontal crack near a rough TC / BC interface; the horizontal crack would have penetrated into the BC and would have simulated interfacial delamination. In general, ERR increased with crack length and as the crack depth approaches the TC / BC interface. A maximum ERR of $G_{max} = 185 \text{ J/m}^2$ was achieved for a crack length of 160 μm at the TC / BC interface. The ERR along the TC / BC interface decayed slightly for longer lengths yet remained large. In agreement with DIC results, this suggested that sustained crack growth was energetically favorable, and that long cracks would drive gradually toward the TC / BC interface. The notable exception to this trend occurred for short cracks near the TC surface ($d < 150 \text{ }\mu\text{m}$, $L < 50 \text{ }\mu\text{m}$); in this region, the ERR decayed until a critical crack length before ERR monotonically increased. This phenomenon was explained by shear lag, as material immediately adjacent to the vertical crack flanks would experience low stress. The primary implication of this “ERR valley” was that growth of short, near-surface delamination cracks would be arrested, even if they initially possessed the most favorable ERR. Beyond this point, growth of cracks closer to the TC / BC interface would dominate (Fig. 10b).

5. Discussion

5.1. Crack formation mechanisms

In-situ observation of the crack formation with DIC revealed the vertical cracks in the TC

formed near the top surface and grew toward the BC interface, driven by tensile stress during the initial heating. Vertical crack initiation and growth in subsequent thermal cycles was minimal due to stress relaxation effects. Some vertical cracks did not reach either the top surface or BC interface, suggesting that the vertical cracks initiated on the interior of the surface. This observation was critical to understanding the formation mechanism of vertical cracks, which has not previously been reported. For instance, vertical cracks were previously modeled as initiating on the surface [36]; with these results in mind, the term “surface cracks” may misrepresent the actual formation mechanism.

The vertical crack path was jagged, indicating that the crack deflected around many obstacles within the TC. Crack branching at these locations gave rise to horizontal cracks, as most of the horizontal cracks initiated at sharp deflections in the vertical crack. It should be noted that many microstructural features, such as inter-splat boundaries, had a distinct horizontal orientation, which would facilitate both vertical crack deflection and branching along the horizontal interface, and FE simulations revealed that small flaws adjacent to the vertical crack could produce favorable ERR for crack growth through almost the entire TC thickness. This mechanism explained the simultaneous formation of vertical and horizontal crack in DIC images during the first heating cycle, as well as in SEM images after only four cycles. This formation mechanism is especially relevant for DVC TBCs, which are typically deposited with large APS passage thicknesses to increase vertical crack density [37], resulting in a microstructure that contains several prominent horizontal interfaces. These interfaces still exist in conventional APS coatings, but the sparsity of vertical cracks may inhibit lateral crack formation at these locations.

5.2. *Crack propagation mechanisms*

SEM inspection of the lateral cracks showed a complex crack growth pattern

characterized by extensive deflection, crack bridging, and crack shielding, which can be explained by the TC microstructure and abundance of pre-existing flaws. When the lateral crack encountered an obstacle, elevated crack-tip stresses enabled crack deflection, as well as growth along weak interfaces adjacent to or in front of the crack tip. This resulted in a network of many horizontal cracks along weak interfaces, as well as the segmented crack growth observed in Figs. 7e and 8. This mechanism is also key to interpreting the FE crack growth results. While the FE-computed ERR for short cracks was generally below G_c obtained by nanoindentation, the pervasive lateral crack propagation indicated that the fracture toughness along weak interfaces was substantially lower. We note that the nanoindentation experiments targeted dense TC grains, such that fracture toughness along porous interfaces could be substantially lower.

Importantly, the FE simulations predicted a region associated with short lateral cracks near the top surface (small d and L) where crack growth would become arrested. The primary implication of this result was that propagation was not favorable for all lateral cracks. This was supported by DIC strain maps in Fig. 7, which revealed that crack growth was most prominent near the TC / BC interface, even though several short cracks near the top surface were apparent in earlier DIC strain maps and SEM images. Second, the simulation results supported the direction of crack growth, which was observed to angle in the direction of highest ERR toward the TC / BC interface. Upon reaching the interface, failure by conventional delamination mechanisms may be expected.

6. Conclusions

New fundamental failure mechanisms of DVC TBCs at temperatures up to 1200°C were captured *in-situ* using high-temperature DIC and FE fracture mechanics analyses, which revealed the coupled formation of vertical and horizontal cracks based on microstructural crack-deflecting

features. Crack initiation was observed at low temperatures during heating from a stress-free state, while crack propagation occurred after cooling from a stress-free state achieved via stress relaxation. Horizontal cracks originated at locations of vertical crack deflection throughout the TC layer, indicating that crack branching at these features contributed to horizontal crack formation. In contrast, lateral crack propagation strongly favored the lower TC region near the BC interface and was found to occur through crack deflection, crack bridging and crack shielding mechanisms. High-throughput FE fracture simulations revealed favorable energy release rates for lateral crack initiation near the TC surface as well as near the BC interface, and stable crack propagation toward the BC interface, which agreed with experimental results. Upon reaching the BC, crack propagation along the interface ultimately leads to coating delamination. This mechanism is unique from conventional delamination failures, in which damage occurs primarily along the interface.

Acknowledgements

This material is based upon work supported by the National Science Foundation (NSF) Graduate Research Fellowship Program under Grant No. 1315231.

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High-Temperature Delamination Mechanisms of Thermal Barrier Coatings: In-situ Digital Image Correlation and Finite Element Analyses

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Graphical Abstract

Fig. 1. Schematic of new coating failure mechanism. (a) After coating deposition, the coating is crack-free. (b) During the first heating cycle, vertical cracks form near “valleys” in the TC / BC interface geometry, and (c) horizontal cracks are found to branch off of the vertical crack. (d) During subsequent heating, the horizontal cracks propagate and lead to coating delamination.

Fig. 2: Nanoindentation results (a) Stiffness of TBC materials was probed at temperatures up to 1000°C (based partially on [18]). (b) The modulus distribution across the TBC cross-section at 25°C and 1000°C.

Fig. 3. Overview of FE mesh geometry (a) Overview of FE mesh with detail of interface geometry. (b) Detail of FE mesh with vertical and horizontal cracks. Note the local mesh refinement around the crack tip. (c) Detail of FE mesh with a longer crack, and flat interface geometry. (d) Summary of fracture analysis parameters in this analysis. Scale bars: (a) 250 μm , (b-c) 50 μm .

Fig. 4: Vertical crack formation in 1100°C sample. (a) Micrograph of TBC sample before heating, showing two vertical proto-cracks (marked by arrows). (b) Plot of temperature-corrected ϵ_{xx} at 600°C, showing early formation of DVCs. (c) Plot of temperature-corrected ϵ_{xx} at 1100°C. (d) Nominal σ_{xx} distribution in TBC due to thermal expansion mismatch during heating [18]. All strains are “temperature corrected” by subtracting thermal expansion. Scale bar: 100 μm

Fig. 5: Horizontal crack formation in 1100°C sample. (a) Optical micrograph of TC before heating. (b) Plot of temperature-corrected ϵ_{yy} at 600°C, showing early formation of horizontal cracks. (c) Plot of temperature-corrected ϵ_{yy} at 1100°C (d) SEM image of highlighted region in (c), showing horizontal crack. Scale bars: (a-c) 150 μm , (d) 20 μm .

Fig. 6: Evidence for temperature-dependent transition between “Crack Initiation” and “Crack Propagation” phases. (a) Median ϵ_{xx} strain as function of annealing time. (b) Median ϵ_{xx} and ϵ_{yy} measured over 80 cycles to temperature of 1200°C

Fig. 7: Thermal cycling to an annealing temperature of 1200°C prompted the growth of lateral cracking, indicated by tensile strain ($\epsilon_{yy} > 0.0$, in yellow) within the top coat. (a) Early crack growth was located just below the surface of the top coat; however, (b) by 10 cycles, prominent crack growth was observed in the middle of the top coat, progressing through (c) 20 and (d) 60 cycles. (e) Horizontal cracks highlighted in green after 80 cycles. Scale bar: 150 μm .

Fig. 8: SEM backscatter images showing the formation of vertical and horizontal TC cracks after four thermal cycles. Horizontal cracks are highlighted in green, and their initiation sites on a vertical crack are marked by red arrows. Selected crack-propagation features are labeled. (a) 1000°C specimen, showing distribution of vertical cracks and large spallation associated with horizontal crack formation (red arrow). (b-g) Detail of horizontal cracking in various specimens: (b-c) 1000°C, (d) 1050°C, (e) 1100°C, (f) 1150°C, (g) 1200°C. (g) Proposed TC cracking mechanism. Scale bars: (a) 250 μm , (b-g) 50 μm .

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Fig. 9. (a-b) Contour plots of total Energy Release Rate ($G_T, J/m^2$) as function of interface geometry. (a) Experimental interface geometry. (b) Flat interface geometry. (c) G_T at $800^{\circ}C$ as function of crack position, and (d) G_T at $1200^{\circ}C$ as function of crack position. All analyses performed with a $10 \mu m$ crack.

Fig. 10. ERR at $25^{\circ}C$ during crack propagation regime, with $T_{ref} = 1200^{\circ}C$. (a) ERR is presented as a function of crack length and crack position. (b) Schematic illustration of crack arresting mechanism for short near-surface cracks (TC in yellow, BC in green).