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Synchrotron XRD Measurements of Thermal Barrier Coatings Subjected to Loads Representing Operational Conditions of Rotating Gas Turbine Blades

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High-energy synchrotron x-rays were used in this work to monitor the internal strain behavior of Thermal Barrier Coatings (TBC) under thermal gradient and mechanical loading. Tubular specimens made from Nickel based super alloy with a TBC-system applied onto the outer surface by Electron Beam Physical Vapor Deposition were used to allow for cooling of the internal surface of the substrate while heating the external surface during thermal mechanical cycling. The coating system consisted of a Yittria Stabilized Zirconia (YSZ) top coat and a MCrAlY bond coat. Through transmission along with a 2D detector allowed for the 2D strain monitoring of each layer during high temperature operation. Monitoring the micro-strain of each phase within the layers provides insight into their high temperature behavior which can be used to further develop predictive models including evolution of elastic strain as well as creep and plasticity. Obtained results have shown a large variation in strains during ramp up of in-phase thermal and/or mechanical load with a significant tensile strain mismatch between the two prominent phases of the bond coat. The YSZ has displayed residual compressive strains at the bond coat/YSZ interface and maintained some compressive residual strain during high temperature holds. These results give valuable insight into the mechanics of these complex systems under various high temperature conditions.

I. Introduction

Athough Yittria Stabilized Zirconia (YSZ) and MCrAlY bond coats have been used extensively in industry for high temperature components of both power generation and jet engine turbines, further knowledge of how their material phases behave under extreme conditions is still highly sought after for the improvement of Thermal Barrier Coatings (TBC). In-Situ monitoring of the mechanical behavior and lattice structure of these layers under high temperature and mechanical loads can provide a wealth of information on the coating's material behavior and integrity. With advanced characterization technology such as synchrotron x-ray diffraction, it is feasible to monitor internal material behavior under such operational conditions.

A combination of both coating design and loading conditions dictate the susceptibility of various locations within the layers to different failure mechanisms. With the extreme conditions experienced by these multi-layered coatings, large variation of internal stresses is exhibited throughout cyclic conditions. Due to cooling of the substrate, temperature differences across the coating are generated creating strain gradients across the thickness of the coating[1] and can trigger certain failure modes within the coating system[2]. By applying Thermal Gradient Mechanical Fatigue (TGMF) loading conditions, the development of cracks have been observed in the bond coat parallel to the surface beneath the top coat furthering the spallation of the coating[2]. Mechanical loading can also

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have large effects on initiating different failure modes. Studies have shown that increasing tensile loads can cause cracks to propagate perpendicularly from the bond coat/TGO interface[3], whereas under low applied tensile loads delamination is dominating failure behavior[4]. Additionally, combinations of thermally grown oxide (TGO) growth stress, applied tensile axial loading and creep relaxation of the TGO has been shown to initiate these cracks under TGMF loading conditions[5].

High-energy x-ray diffraction (XRD) is a valuable tool in characterizing the material behavior within the internal depths of TBC layers. This method allows for the through penetration of samples to acquire depth resolved XRD measurements. High-energy XRD has been used to display how the in-plane and out-of-plane strains are affected by application process[6]. Synchrotron XRD has also been used for various ex-situ residual strain studies of cycled TBC samples providing for validation of theoretical models[7, 8]. By applying *in-situ* thermal and mechanical conditions, tensile loads have shown to develop a tensile state of stress in the TGO of as-coated specimens at high temperature[9].

Substantiating the link between in-situ measured strains under operational conditions and locations of different failure modes, will further the application field of TBCs. Both micro and macro strains within the depth of the layers are vital to the improvement of material models to predict the stress evolution leading to failure and in turn improve the coating application design process. The ability to track the micro-strain exhibited within the grains of the individual phases is important for multi-phase coatings such as TBCs. Initially MCrAlY bond coats are comprised primarily of β-NiAl and γ-Ni phases[10]. The YSZ top coat also contains multiple phases as the tetragonal t'-YSZ transforms into the cubic, tetragonal t, and monoclinic phases after extended thermal exposure[11]. Understanding the progression of the micro-strain in the individual phases during loading gives a better understanding of the global properties of these layers such as yield strength and fracture toughness.

This work will present the measured strain within the phases of as-coated EB-PVD specimens subjected to thermal gradient and mechanical load. To represent cyclic service conditions, 80 minute cycles were applied while monitoring the XRD measurement throughout. It improves upon previous *in-situ* strain work with uniform thermal conditions by developing techniques which can apply thermal gradients across the coating while measuring strain in the curvature of the coatings. Strains in each phase within the bond coat and YSZ are displayed for a cycle with a maximal applied nominal stress of 64 MPa and an estimated 115 °C temperature drop across the YSZ top coat.

II. Experimental

The samples, which were manufactured at the German Aerospace Center (DLR), comprised IN100 tubular substrates with 8mm outside diameter and 4 mm inside diameter and a TBC system applied via Electron Beam Physical Vapor Deposition (EB-PVD). The as-coated TBC on the sample consisted of 7-8 wt% Yttria-Stabilized Zirconia (YSZ) ceramic topcoat with a thickness of 211 +/- 4 µm and a NiCoCrAlY bond coat with a thickness of 118 +/- 4 µm. The specimens were manufactured to a tubular shape to allow for the application of a temperature gradient across the coating thickness by cooling the internal surface with pressurized air and heating the external surface by means of a radiation heater. The outer coating surface was heated up to 1000 °C with a heater containing 4 cylindrical lamps with maximum power output of 2000 Watts each with radiation focused by elliptical mirrors onto the specimen. For experiments studying gradient effects, the mass flow of internal coolant through the inner diameter of the specimen substrate was controlled. To represent a service cycle, the external sample temperature was ramped up to of 1000 °C throughout a 20 minute duration and then held for 40 minutes before ramping back down. An applied nominal mechanical stress of 64 MPa and internal coolant flow of 75 SLPM were held constant throughout the cycle. Through transmission XRD measurements were taken repeatedly during the entire cycle by grazing tangentially to the coating layers. Further detail on the experimental setup is described in previous work[12].

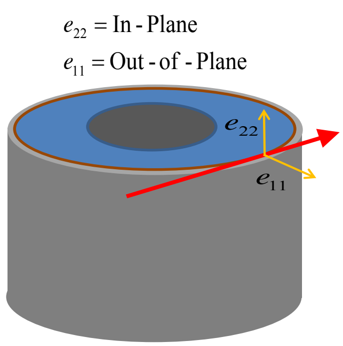

Figure 1. Beam Placement

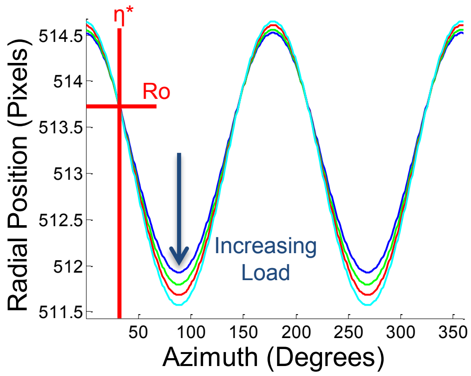

Figure 2. Reference Azimuth for Strain Calculation

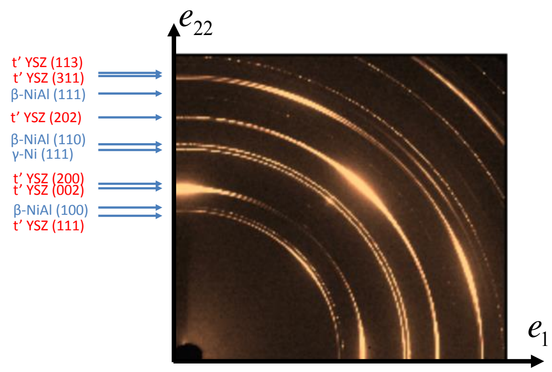

Figure 3. XRD Phase Results

Measurements were taken at the 1-ID beamline at the Argonne National Laboratory Advanced Photon Source with beam energy capable of up to 100 keV. For these experiments, 65 keV was used for through penetration of the sample tangential to the layers, as shown in Figure 1, with a beam width of 30 µm and height of 300 µm. At each measurement time, a scan of 10 windows was taken spanning through the coating layers. Each collection was taken by a high resolution 200 µm pixel size 2D detector, providing diffraction rings for lattice planes of each phase as low as 1.29 Angstroms. Lower lattice planes were limited by the 1 inch diameter window size exiting the heater.

III. Data Calculation

With a 2D detector, diffraction measurements can be taken around a 360^0 azimuthal angle on a plane parallel to the e11-e22 plane. This allows for the measurement of radial deviation of the diffraction ring, which results in an ellipse under strained conditions and provides 2D strain calculations. This radial deviation is determined relative to a strain invariant radius R_o which is referenced at a strain free azimuth angle, η^*, on the measurement plane between the e11 and e22 axis. This strain invariant reference angle, as displayed in Figure 2, is determined by comparison of the radial curves measured during varying applied tensile loads[13]. With measurements of radial position of diffraction rings, various material characteristics can be determined from the calculated d-spacing of the respective lattice plane.

IV. Results

Shown in Figure 3, is an XRD measurement taken at a location near the inner bond coat radius. Due to the circular shape of the coating, phases are obtained for both bond coat and YSZ at this location. The bond coat predominantly consists of two phases which are the β-NiAl phase and a solid solution γ-Ni phase. Also present, is a textured γ '-Ni_3Al phase which provides signals of considerably smaller intensity than the β-NiAl and γ-Ni phase. The YSZ consisted primarily of a tetragonal t'-YSZ phase.

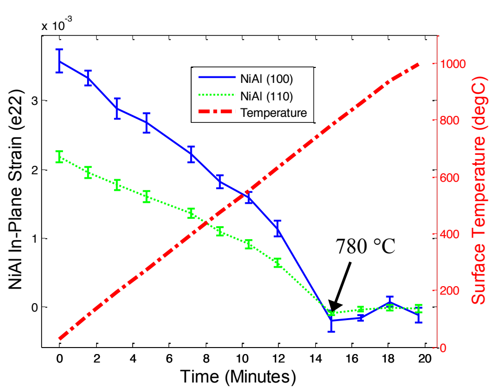

Figure 4. NiAl (100) and (110) e22 Strain

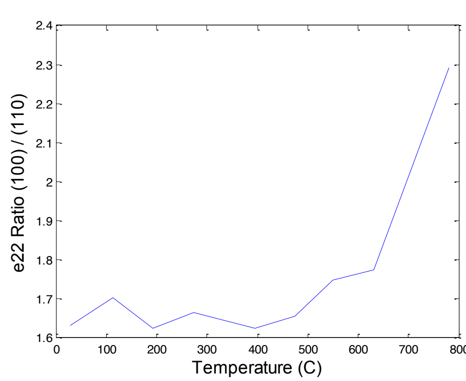

Figure 5. NiAl Anisotropy (100)/(110) Strain Ratio

The strains were calculated throughout the entire cycle with a nominal applied stress of 64 MPa and a coolant flow of 75 standard liters per minute (SLPM). Through estimation using the d-spacing measurement across the YSZ thickness along with measured thermal expansion coefficients, a 75 SLPM flow rate corresponds to a temperature drop of roughly 115 ^{0}C across the YSZ layer. The results displayed here, show the strain for single locations in both the bond coat and YSZ near the interface. Since this is a grazing method, the two strains displayed are radial (out-of-plane) and axial (in-plane), which are represented as e11 and e22 respectively. The in-plane strain is the most prominent due to the thermal expansion mismatch between the layers in that direction. The strain measurements from XRD provide a measurement of anisotropy of the phases due to the fact that it is a micro-strain measurement. While a phase such as NiAl is very anisotropic within a grain, the bond coat is essentially isotropic on the macro scale due to the fine grained nature of the as-coated MCrAlY bond coat. Comparing two lattice planes of the NiAl provides a measure of how anisotropic the phase is.

Shown in Figure 4, are the e22 strains for NiAl (100) and (110) during ramp up from room temperature to 1000 ^{o}C. Figure 5 displays the ratio of the strains plotted versus temperature. This corresponds closely to the inverse relationship of the elastic moduli for the two planes which is roughly 1.8 at room temperature determined from measured single crystal constants[14]. Shown in Figure 6 and 7 are the strain response of the β-NiAl (100) and γ-Ni (111) phases respectively. This displays that the bond coat in-plane strain is highly in tension at room temperature indicating that the thermal expansion coefficient for the bond coat is significantly higher than for the IN-100 substrate. As the surface temperature is ramped up to 1000 ^{o}C, which corresponds to the coating application temperature, the residual strain moves towards zero. It can be seen that the bond coat strain also converges to zero when the outer surface temperature reaches just 800 ^{o}C. This is likely due to creep experienced in the bond coat before high temperature is reached. Displayed in Figure 8 is the t-YSZ (111) strain throughout the same cycle. The YSZ is shown to be in compression for the in-plane direction at room temperature while also moving towards zero at high temperature while maintaining some residual strain.

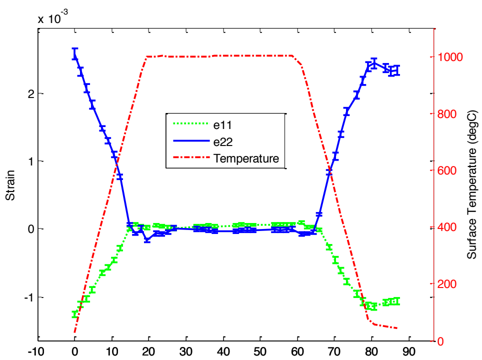

Figure 6. β-NiAl (110) Strain Under 64 MPa Applied Nominal Stress and 75% of Maximal Thermal Gradient

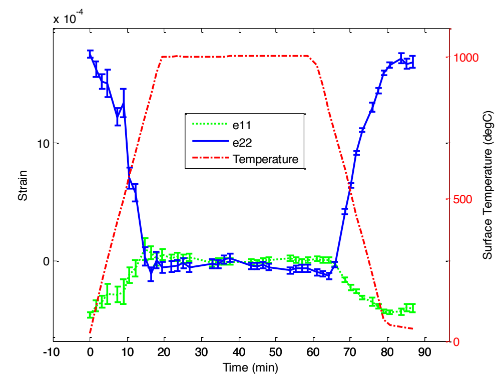

Figure 7. γ-Ni (111) Strain Under at 64 MPa Applied Nominal Stress and 75% of Maximal Thermal Gradient

V. Conclusion

In this study strain for the β-NiAl, γ-Ni, and t'-YSZ phases near the bond coat/YSZ interface were monitored throughout a thermal cycle with 64 MPa applied nominal mechanical stress and a 115 ^{o}C temperature drop across the YSZ. This has displayed the residual strains in each phase of the as-coated specimen as they move towards zero at the cycle temperature which corresponds to the manufacturing temperature. Both bond coat phases display an early convergence of the strain to zero before the YSZ temperature achieves its max temperature suggesting that the bond coat has a nonlinear strain response to any further applied stress. Results such as these for as-coated specimens

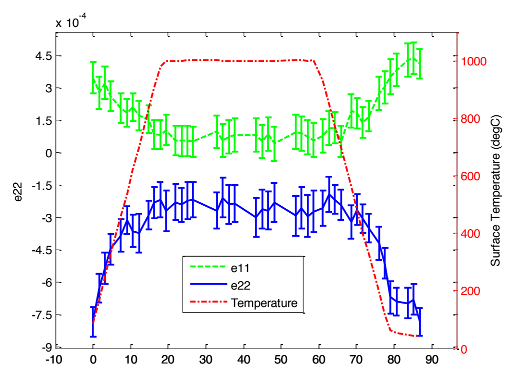

Figure 8. YSZ (111) Strain Under 64 MPa Applied nominal stress and 75% of maximal Thermal Gradient

along with fatigued specimens will give valuable information on how the strain evolves at high temperature throughout a coating lifetime. Creating validated material models with this information will work to achieve the full potential of these coatings.

Acknowledgments

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